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Evaluation of a Multichannel Inductively Coupled Plasma-Optical Emission Spectrometer Modified to Minimize and Correct Scattered Light Effects

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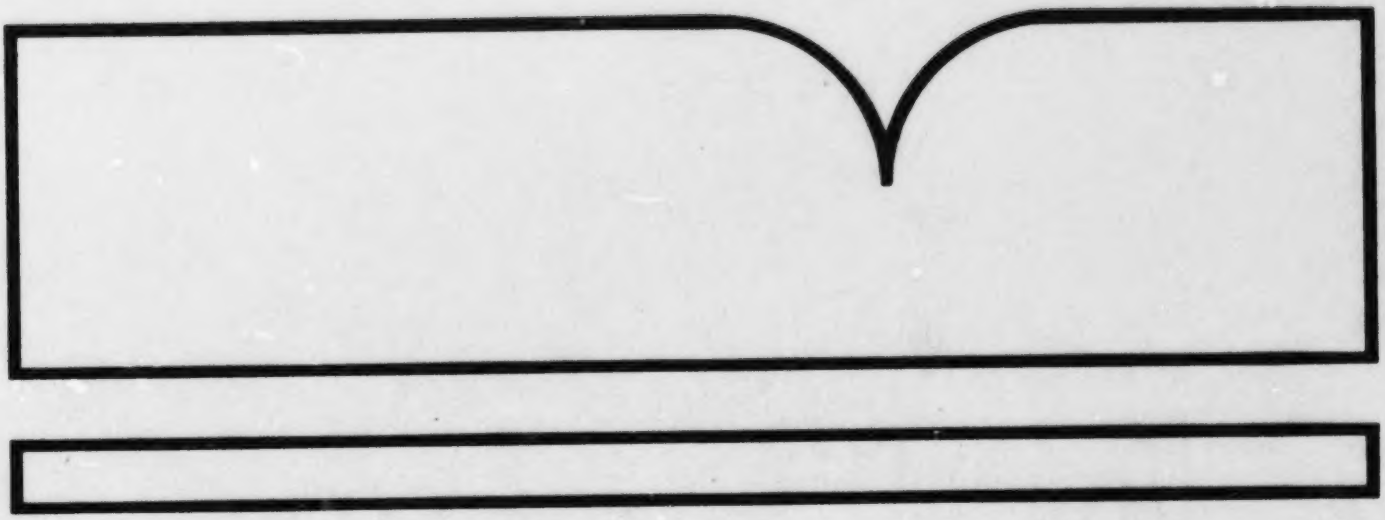

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16. ABSTRACT
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Evaluation of a Multichannel Inductively Coupled Plasma-Optical Emission Spectrometer Modified to Minimize and Correct Scattered Light Effects

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In a study of an inductively coupled plasma optical emission spectrometer, data from an early commercially available instrument are compared with data from the same instrument after modifications to correct observed inadequacies. Results show negligible changes in powers of detection but considerable reductions in scattered light interferences following the installation of a holographic grating.

Index Headings: **Emission spectroscopy; Inductively coupled plasma; Elemental analysis.**

INTRODUCTION

Inductively coupled plasma-optical emission spectrometry (ICP-OES) is an effective technique for rapid trace multielement analyses in a variety of matrices.[1-4] One of the limiting factors for detection of trace elements using this technique, however, is the magnitude of interference correction required to produce valid data. Certainly if only minor interference corrections are required, the contribution of errors due to multiple measurements can be sharply reduced or eliminated. Data shown in this article will indicate the efficiency of these modifications in improving analytical data.

Spectral interferences in ICP-OES analysis are generally referred to as either direct spectral or scattered light. Direct spectral interferences occur when two or more elements present in a sample emit spectra that cannot be resolved by the instrument optical system. Scattered light interferences are caused by grating imperfections or by internal surfaces that act as reflectors. This scattered light is then ultimately interpreted as analyte signal by the detector. Direct spectral interferences can be identified through the use of tables of spectral intensities[5-7] and by analyzing pure standard material. These direct spectral interferences can then be quantified and the data used to correct the analysis of unknowns. Scattered light interferences are not quite so easily resolved because diffraction grating imperfections and the array of analyte lines will vary among individual spectrometers.[8,9] False spectral lines due to periodic errors in grove spacings on diffraction gratings are referred to as Rowland or Lyman ghosts and can also contribute to interference. The amount of unwanted light due to ghosts is dependent

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upon the quality of the diffraction grating. Grating imperfections called satellites are false spectrum lines that usually are found near parent lines and are due to misplaced grooves. In poor quality gratings, satellites will cause much scattered light, which is generally referred to as grass. The amount and origin of the scattered light will vary with the age of the ICP-OES unit. Early systems will probably exhibit greater scattered light problems than the models of instruments currently commercially available.

The user of the ICP-OES should first minimize scattered light regardless of its origin. Any remaining light must ultimately be quantitatively determined so valid corrections can be applied. Numerous investigators have reported modifications and changes made to ICP-OES units to correct or reduce scattered light of various origins.[1-4, 9, 10] Some[9] suggest use of higher quality diffraction gratings, flat black coatings of nonoptical surfaces, baffles to trap zero and other order radiations not used in the instrument array, and light tunnels in the secondary optics system.

In this report, corrective modifications to minimize scattered light in the ICP-OES will be discussed. The data presented will be representative of conditions of the ICP-OES before and after modifications were made to the instrument. The ICP-OES has been evaluated with a mechanically[11] ruled grating having 1920 grooves/mm, a holographically[12] recorded grating having 1920 grooves/mm, and narrow band pass interference filters[13] on detectors at wavelengths ≤250 nm.

TABLE I. Spectrometer line arrays.

Original		Present	
Element[a]	Wavelength (nm)	Element[a]	Wavelength (nm)
Ag I	328.07	Ag I	328.07
Al I	308.22	Al I	308.22
Al I	396.15	As I[b]	193.70
As I	193.70	B I[b]	249.68
B I	249.68	Ba II	455.40
BaII	455.40	Be II	313.04
Be II	313.04	Bi I[b]	223.06
C I	247.86	Ca II	315.89
Ca II	315.89	Cd II[b]	226.50
Cd II	226.50	Co II[b]	228.62
Co II	238.89	Cr II	267.72
Cr II	267.72	Cu I	324.75
Cu I	324.75	Fe II	259.94
Fe II	259.94	Hg I	253.65
Hg I	253.65	Mg II	279.08
Mg II	279.55	Mn II	257.61
Mn II	257.61	Mo II	287.15
Mn I	403.08	Na I	330.23
Mo II	287.15	Ni II[b]	231.60
Ni II	231.60	Pb II[c]	220.35
Pb II	220.35	Sb I[b]	206.84
Pb I	405.78	Se I[b]	196.03
Sb I	206.84	Sn I	303.41
Se I	196.03	Sr II	407.77
Sn I	303.41	Te I[b]	214.28
Sr II	407.77	Th II	401.91
Ti II	334.90	Ti II	368.52
V II	292.40	Tl I	351.92
Y II	242.22	V II	292.40
Y II	371.03	Y II	371.03
Zn I	213.86	Zn I[b]	213.86

[a] I denotes radiation originating from neutral atom; II denotes radiation originating from singly ionized atoms.
[b] Narrow band pass filters installed on detector.
[c] Solar blind photomultiplier tube installed.
NOTE: In the present array, Bi, Co, and Te are second order.

TABLE II. Spectrometer desorption and operating conditions.

Basic spectrometer and data system	Inductively coupled plasma
Mount: 1.0 m Paschen-Runge, f30	Generator: Air cooled, 3 kW rating, continuous
Slits: 12 μm entrance, 50 μm exit, all wavelengths	Frequency: 27.1 MHz, crystal controlled
Grating: 1920 rulings/mm, holographically recorded diffraction grating. Grating specifications shown in Table III.	Power: 1600 ± 50 W forward power, 2 W reflected, 150 W coupling loss
Observation height: 17 mm above the rf coil (vertical aperture defines range of 15–19 mm)	Regulation: To forward power, 1.0%
Readout: PDP 11/05, 16K words, computer with DecWriter console and floppy disk data and program storage; BLISS software, (BASIC language integrated software systems); successively segmented third-order regression defining calibration curves; second-order regression defining interference corrections on a concentration basis.	Coil: Two turn copper, water cooled
	Impedance matching network: Series-parallel capacitance
	Torch: Integral, quartz, 18 mm o.d., three concentric tube configuration
	Gas: Argon
	Coolant: 10.5 liters/min
	Plasma: 1.5 liters/min
	Aerosol carrier: 1.0 liter/min
	Gas flow control: Three stage regulation of pressure, capillary restrictors, interlocked
	Nebulizer: All glass, permanently aligned, coaxial pneumatic, computer controlled de-salter

I. EXPERIMENTAL

A. Instrumentation. The ICP-OES used in this study was in a QA-137 direct-reading polychromater with an inductively coupled argon plasma source which was a standard commercially available instrument in 1975.[11] The torch, spray chamber, and pneumatic nebulizer system were all of standard configuration and equipment.[14] Some minor modifications made to this ICP-OES have been reported.[13] After the initial evaluation of the ICP-OES, the instrument was updated in 1978 by the vendor[11] with some current state-of-the-art modifications and equipment. The mechanically ruled diffraction grating was replaced with a holographically recorded diffraction grating, and the elements bismuth, sodium, tellurium, thorium, and thallium were added to the array. To accommodate the addition of these five elements, aluminum (396.15 nm), carbon (247.86 nm), manganese (403.08 nm), lead (405.78 nm), and yttrium (242.22 nm) lines were eliminated. Narrow band pass interference filters were placed on those lines indicated in Table I and lead (220.35 nm) was equipped with a solar blind photomultiplier detector. An automated scanning primary slit was installed to facilitate the use of the polychromator as a scanning device. The existing data system was updated with sufficient core and memory to handle the ICPQ-BLISS software system.[11] The ICPQ-BLISS software facilitates instrument control and handles data acquisition, data background and interference corrections, data storage, and a digital plotter routine to output scan data. Operating conditions and the basic spectrometer for this equipment are described in Table II and holographic grating specifications are shown in Table III.

B. Reagents and Materials. Distilled-deionized water was used for sample preparations and rinsing procedures. Standard reference solutions were purchased from Spex Industries, Inc., Metuchen, NJ, and National Spectrographic Labs, Inc., Cleveland, OH. Some laboratory standard solutions were made starting with metals or inorganic compounds purchased from Spex Industries. Nitric acid (Ultrex) was purchased from J. T. Baker, Phillipsburg, NJ.

II. RESULTS AND DISCUSSION

A. Powers of Detection. In this section, representative data and spectra are shown to indicate initial and present qualities of data using this ICP-OES (Table IV). The first column (Initial) shows data taken on the original ICP-OES; the second column (Present) shows data taken on the same ICP-OES after modification. The data indicate that the modifications to the ICP-OES did not have a deleterious effect on the powers of detection based on tests using single element standard solutions with 1% HNO_3 under multielement analysis compromise instrument conditions.

B. Scattered Light Interference. When this ICP-OES was originally put into use, scattered light generated by both calcium and magnesium was a major problem when environmental samples were analyzed. Most scattered light in this ICP-OES was found to originate at the mechanically ruled grating and to a lesser extent from reflective surfaces in the spectrometer.[14] The baffling previously reported[14] has been replaced by the vendor with a dual purpose slit and/or interference filter mount assembly at the detectors. As can be seen in Table V, some scattered light equivalents due to 500 μg/ml of calcium have been significantly reduced by the modifications to the ICP-OES. Arsenic shows a factor of 95 times less interference due to calcium scattered light; others such as Hg, Pb, Sb, Se, and Sn have been significantly improved in interfering scattered light characteristics. Scattered light effects due to 100 μg/ml of magnesium are shown in Table VI. Arsenic and selenium show most significantly the decreases in amounts of scattered light due to magnesium. The data in Table VII show scattered light equivalents due to calcium and magnesium at 1000 μg/ml. Examination of Tables V, VI, and VII shows clearly that the problems of scattered light in the original instrument have been significantly reduced by the modifications of the ICP-OES.

TABLE III. Holographic grating specifications.

Blank	
Fused quartz or cervit	
80.0 ± 0.2 mm diameter	
10.0 ± 0.2 mm thick (at edge)	
997.9 mm ± 0.5 mm radius of curvature	
Bevel on ruled side acceptable (e.g., 45° with 1.5 mm width maximum)	
Aluminum coating	
Rulings	
Full aperture of grating	
1920.21 ± 0.02 grooves/mm	
Performance specifications	
Optical efficiency:	40% from 1900 to 4600 A
Optical resolution:	85% of theoretical
Ghost intensity:	10^{-12} of parent line
Optical scatter;	10^{-7} of parent line 10 A from parent line
	10^{-8} of parent line 25 A from parent line
	10^{-9} of parent line 50 A from parent line
Speed uniformity:	The speed of the left, center, or right ⅓ area shall not differ from any other ⅓ by more than 25%.

TABLE IV. Powers of detection.[a]

Element	Wavelength (nm)	Initial (μg/ml)	Present (μg/ml)
Ag	328.07	0.002	0.002
Al	308.21	0.040	0.036
As	193.70	0.040	0.042
B	249.68	0.004	0.006
Ba	455.40	0.0002	0.00006
Be	313.04	0.0004	0.0003
Bi[b]	223.06	...[c]	0.054
Ca	315.89	0.008	0.004
Cd	226.50	0.003	0.002
Co[b]	228.62	0.003	0.004
Cr	267.72	0.003	0.002
Cu	324.75	0.001	0.001
Fe	259.94	0.001	0.001
Hg	253.65	0.010	0.009
Mg	279.08	...[c]	0.021
Mn	257.61	0.0002	0.0004
Mo	287.15	0.006	0.009
Na	330.23	...[c]	0.67
Ni	231.60	0.007	0.007
Pb	220.35	0.020	0.022
Sb	206.84	0.020	0.020
Se	196.03	0.060	0.062
Sn	303.41	0.050	0.058
Sr	407.77	0.0003	0.0001
Te[b]	214.28	...[c]	0.033
Th	401.91	...[c]	0.003
Tl	368.52	...[c]	0.003
Tl	351.92	...[c]	0.050
V	292.40	0.002	0.001
Y	371.03	0.0003	0.00009
Zn	213.86	0.004	0.005

[a] The analyte concentration required to produce a signal 2 times the standard deviation at the analytical line.
[b] Only second order lines used narrow band pass filter.
[c] Line did not exist for this element in original array.

C. Wavelength Scans Showing Scattered Light Interference. A computer-controlled stepping motor on the primary slit of the ICP-OES allows simultaneous wavelength scans to be made at each detector of the polychromator. These wavelength scans can be used by the operator to observe the area around the analyte signal of interest. The wavelength profiles will warn the operator when background shifts occur or when spectral interferences in unknown sample matrices require more than a normal blank correction to produce valid analytical data. The stepping motor assembly can also be used to produce analytical data. In the analytical mode of operation, the operator selects two points for background corrections that will result in proper background subtraction at the analytes of his choice. Background corrections such as these are most necessary for valid analyses in complex sample matrices.

In Fig. 1, plots of data show scattered light characteristics in the ICP-OES when originally purchased.[13] Significant changes are apparent in the spectra at the nickel 231.6 nm line when calcium and magnesium are present in the sample. If a normal blank procedure were applied

TABLE V. Scattered light equivalents due to calcium at 500 μg/ml.

Element	Wavelength (nm)	Scattered light equivalents	
		Initial (μg/ml)	Present (μg/ml)
Ag[a]	328.07	...	0.0014
Al	308.22	0.0404	0.000
Al	396.15	1.1459	...[b]
As	193.70	2.9367	0.031
B	249.68	0.0242	0.0011
Ba	455.40	0.0047	0.0000
Be	313.04	0.0004	0.0000
Cd	226.50	0.0406	0.0015
Co	238.89	0.0426	...[b]
Cr	267.72	0.0292	0.0000
Cu	324.75	0.0027	0.0010
Fe	259.94	0.0094	0.0000
Hg	253.65	0.1380	0.0000
Mn	257.61	0.0012	0.0000
Mn	403.08	0.0441	...[b]
Mo	287.15	0.0719	0.0000
Ni	231.60	0.0933	0.0000
Pb	220.35	0.5629	0.0000
Pb	405.78	0.6355	0.0000
Sb	206.84	0.5217	0.0000
Se	196.03	1.3211	0.0000
Sn	303.41	0.6478	0.0281
Sr	407.77	0.0564	Impurity in standard material
Ti	334.90	0.0391	...[b]
V	292.40	0.0094	0.0006
Y	242.22	0.1329	...[b]
Y	371.03	0.0022	0.0006
Zn	213.86	0.0320	0.0000
Bi	223.06	...[b]	0.0000
Co	228.62	...[b]	0.0000
Na	330.23	...[b]	0.0000
Te	214.28	...[b]	0.0000
Th[c]	401.91	...[b]	0.0782
Ti	368.52	...[b]	0.0038
Tl	351.92	...[b]	0.0000

[a] Data not available.
[b] Line not in array.
[c] Overlapping spectral interference of nearby calcium spectra.

TABLE VI. Scattered light equivalents due to magnesium at 100 μg/ml.

Element	Wavelength (nm)	Scattered light equivalents	
		Initial (μg/ml)	Present (μg/ml)
Ag[a]	328.07	...	0.0000
Al	308.44	0.0047	0.0000
Al	396.15	−0.0010	...[b]
As	193.70	0.3544	0.0000
B	249.68	0.0025	0.0012
Ba	455.40	0.0006	0.0000
Be	313.04	0.0002	0.0000
Ca	315.89	−0.0062	0.0012
Cd	226.50	0.0052	0.0000
Co	238.89	0.0052	...[b]
Cr	267.72	0.0015	0.0000
Cu	324.75	−0.0003	0.0000
Fe	259.94	0.0041	0.0000
Hg	253.65	0.0365	0.0000
Mn	257.61	0.0006	0.0000
Mn	403.08	−0.0031	...[b]
Mo	287.15	0.0386	0.0051
Ni	231.60	0.0167	0.0000
Pb	220.35	0.0506	0.0000
Pb	405.78	0.2910	...[b]
Sb	206.84	0.0703	0.0000
Se	196.03	0.1777	0.0000
Sn	303.41	−0.0197	0.0000
Sr	407.77	0.0001	0.0000
Ti	334.90	−0.0029	...[b]
V	292.40	0.0010	0.0007
Y	242.22	0.0131	...[b]
Y	371.03	0.0010	0.0000
Zn	213.86	0.0139	0.0000
Bi	223.06	...[b]	0.0000
Co	228.62	...[b]	0.0000
Na	330.23	...[b]	0.0000
Te	214.28	...[b]	0.0000
Th	401.91	...[b]	0.0000
Ti	368.52	...[b]	0.0000
Tl	351.92	...[b]	0.0000

[a] Data not available.
[b] Lines not in array.

using deionized water, the analysis for nickel in this sample containing Ni, Ca, and Mg would have an error of ~+40%. Shown in Fig. 2 are data taken at the Ni 231.6 nm line after the ICP-OES had been modified as discussed in section I of this report. The plots of data shown (Fig. 2), are of Ca at 1000 μg/ml (C), Mg 1000 μg/ml (M), and deionized water as the blank (B). Dial division #506 corresponds to the Ni 231.6 nm line. The data in Figs. 1 and 2 indicate the change in scattered light characteristics of this ICP-OES after modification. Fig. 2 shows no difference in intensity at 231.6 nm for the three solutions noted indicating no interference due to Ca or Mg at 231.6 nm. Data shown in Fig. 3 were recorded after removal of the narrow band pass filter from the detectors. Plots in Fig. 3 represent data of Ca at 1000 μg/ml (*), Mg at 1000 μg/ml ($), Ni at 0.5 μg/ml (@), and deionized water (B). No differences in the data or levels of scattered light interferences at these concentrations with or without the interference filter are apparent. These findings are consistent with those shown in Tables V, VI, and VII where data are presented in terms of scattered light equivalents. Fig. 4 shows data taken in the area of Pb 220.4 nm on the ICP-OES in the original configuration. Scattered light from the Ca at 150 μg/ml and Mg at 40 μg/ml solution easily overshadow any signal of Pb at 0.05 μg/ml in the early instrument configuration. Fig. 5 shows data in the same spectral region as that of Fig. 4, Pb 220.4 nm. The plotted data in Fig. 5 using the modified ICP-OES are of Ca at 1000 μg/ml (C), Mg at 1000 μg/ml (M), and deionized water (B). The detector in the modified instrument configuration for the Pb 220.4 nm line is a solar blind photomultiplier tube. As with Ni at 231.6 nm, much higher concentrations of calcium and magnesium can be introduced into the plasma of the modified ICP-OES without encountering the same scattered light interferences of the early instrument.

III. CONCLUSIONS

The primary intent of the modification to this ICP-OES was to minimize scattered light interference and secondly to properly correct for any remaining sources of interference. Certainly the data presented and discussed in this paper show significant minimizing of scattered light interferences in the modified ICP-OES system. The reduction of scattered or unwanted light reaching the detectors is shown to result in a large part from the use of a holographic grating. The use of both the holographic

TABLE VII. Scattered light equivalents due to calcium and magnesium at 1000 μg/ml from modified ICP-OES.

Element	Wavelength (nm)	Calcium (μg/ml)	Magnesium (μg/ml)
Ag	328.07	0.0005	0.0007
Al	308.21	0.0000	0.0000
As	193.70	0.0000	0.0000
B	249.68	0.0000	0.0032
Ba	455.40	Impure standard	0.0000
Be	313.04	0.0000	0.0000
Bi	223.06	0.0000	0.0000
Ca	315.89	1000	Impure standard
Cd	226.50	0.0008	0.0019
Co	228.62	0.0005	0.0010
Cr	267.72	0.0000	0.0019
Cu	324.75	0.0000	0.0003
Fe	259.94	0.0000	0.0026
Hg	253.65	0.0000	0.0051
Mg	279.08	0.0008	1000
Mn	257.61	0.0000	0.0000
Mo	287.15	0.0000	0.045
Na	330.23	0.0000	0.0000
Ni	231.60	0.0000	0.0000
Pb	220.35	0.0000	0.0151
Sb	206.84	0.0000	0.0075
Se	196.03	0.0000	0.0354
Sn	303.41	0.0000	0.0155
Sr	407.77	Impure standard	0.0000
Te	214.28	0.0000	0.0000
Th	401.91	6.1361	0.0000
Ti	368.52	0.0038	0.0000
Tl	351.92	0.0000	0.0000
V	292.40	0.0002	0.0023
Y	371.03	0.0003	0.0000
Zn	213.86	0.0000	0.0049

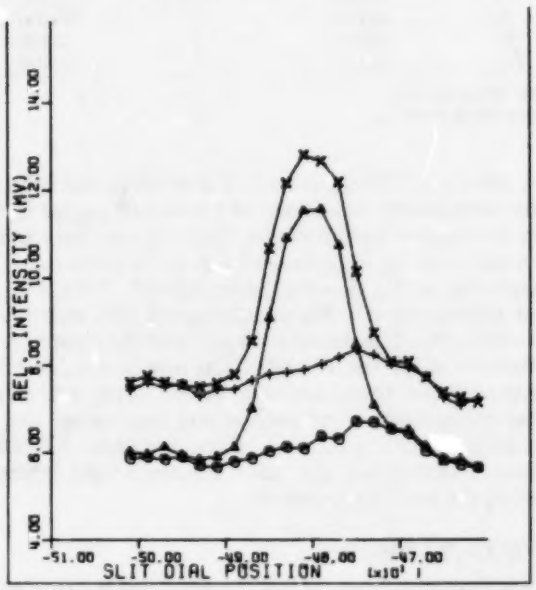

FIG. 1. Wavelength scan showing the effect of Ca 150 μg/ml and Mg 40 μg/ml) on original spectrometer system at Ni 231.6 nm at a concentration of 0.2 μg/ml. ○, deionized water; △, Ni 0.2 μg/ml; ×, Ca 150 μg/ml, Mg 40 μg/ml plus Ni 0.2 μg/ml; +, Ca 150 μg/ml, Mg 40 μg/ml. (From Winge *et al.*[14])

grating and narrow band pass filters at the detectors was shown not to be needed at the concentration levels discussed here. Scattered light interferences in the sec-

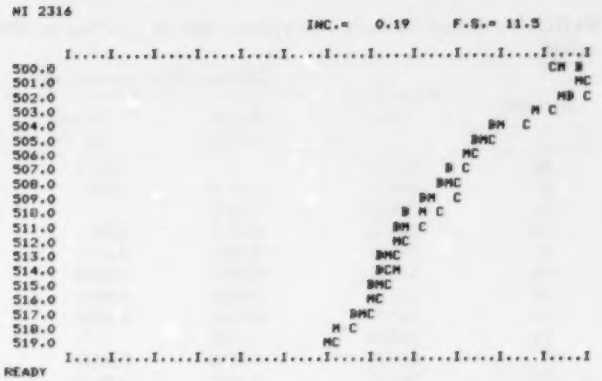

FIG. 2. Wavelength scan showing the effect of Ca 1000 μg/ml and Mg 1000 μg/ml at 231.6 nm (Ni) from modified ICP-OES using narrow band pass filter on Ni detector. C, Ca 1000 μg/ml; M, Mg 1000 μg/ml; B, deionized water 1% HNO_3.

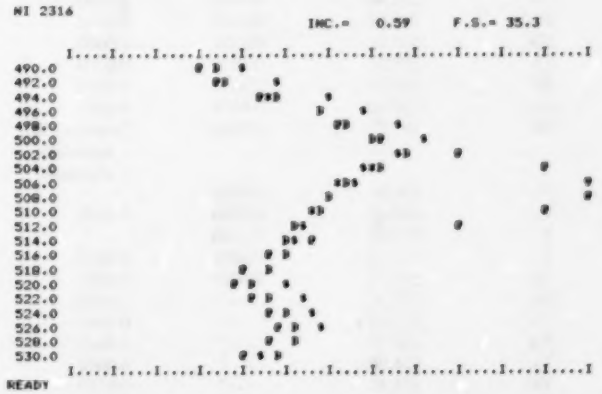

FIG. 3. Wavelength scan showing the effect of Ca 1000 μg/ml and Mg 1000 μg/ml on Ni 231.60 nm from modified ICP-OES after removal of narrow band pass filter. *, Ca 1000 μg/ml; $, Mg 1000 μg/ml; B, deionized water 1% HNO_3; @, Ni 0.5 μg/ml.

ondary optical system of the spectrometer have been reduced with the use of dual purpose slit/filter mount assemblies that cover the detectors.

The proper correction of the remaining scattered and/or unwanted light is now handled more precisely with the addition of a different software and data handling system. The "BLISS" software system uses second order polynomial calculations for interference corrections as compared to linear corrections in the original software delivered with the ICP-OES. Another feature that allows the analyst to apply more accurate and knowledgable corrections for interference is the computer-operated primary slit. The scanning primary slit allows wavelength scans such as those shown in Figs. 1 through 5 of this paper. The software will plot as many as four scans per graph to permit differences in background or interferences to be observed. The scanning slit mechanism also is capable of quantitative analysis and allows the operator to make two-point background corrections for any or all of the programmed element capability of the polychromator system.

When all efforts to minimize scattered or unwanted light and to finally correct for the remaining interferences are considered, the corrective modifications discussed

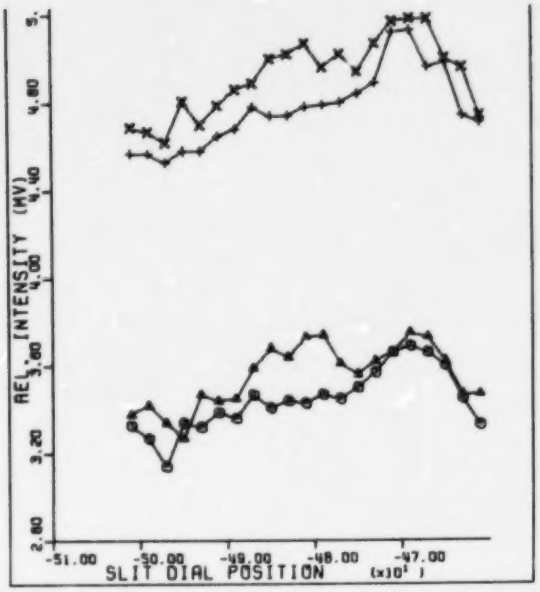

FIG. 4. Wavelength scan showing the effect of Ca 150 μg/ml and Mg 40 μg/ml on original spectrometer system at Pb 220.4 nm at a concentration of 0.05 μg/ml. ○, deionized water; △, 0.05 μg/ml Pb; +, Ca 150 μg/ml and Mg 40 μg/ml; ×, Ca 150 μg/ml, Mg 40 μg/ml plus Pb 0.05 μg/ml. (From Winge *et al.*[14])

here appear to be worthwhile and effective in producing valid analytical results by ICP-OES.

FIG. 5. Wavelength scan showing the effect of Ca 1000 μg/ml and Mg 1000 μg/ml at 220.4 nm (Pb) from modified ICP-OES. Detector is a solar blind photo multiplier. C, Ca; M, Mg; B, Deionized water 1% HNO_3.

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